United States Department of Agriculture
Agricultural Marketing Service, Science & Technology
Pesticide Data Program

SOP No.: PDP-LABOP-9		Page 1 of 8
Title: Laboratory Procedures for Aldicarb/Potatoes Project		
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1. Purpose:

To provide standard procedures for the receipt, preparation, storage, analysis, reporting, and disposal of USDA, AMS Pesticide Data Program (PDP) Aldicarb/Potatoes project samples.

2. Scope:

This standard operating procedure (SOP) shall be followed by all analytical laboratories which are conducting pesticide residue studies for the PDP Aldicarb/Potatoes project.

3. Outline of Procedure:

5.1 Sample Receipt
5.2 Sample Preparation
5.3 Sample Storage
5.4 Sample Analysis and Reporting
5.5 Sample Disposal

4. References:

USDA, AMS PDP Program Plan, July 1997 - March 1998.
Robert Smith, USDA, AMS, S&TD, Residue Branch, Memorandum, January 30, 1997
Ed Zager, EPA, OPPTS, OPP, HED, personal communication, July 23, 1996
Robert Epstein, USDA, AMS, S&TD, Residue Branch Meeting, July 22, 1996

5. Specific Procedures:

These operating procedures provide minimum requirements for the receipt, preparation, storage, analysis, reporting, and disposal of USDA, AMS, PDP Aldicarb/Potatoes project samples. Each participating laboratory shall, as part of their internal laboratory SOPs, have written instructions providing specific details concerning how these procedures have been implemented in that laboratory. Both the USDA, AMS SOPs and the laboratory's internal SOPs shall be used as the measure of compliance in the event of a USDA, AMS laboratory review.

5.1 Sample Receipt

a. Each sample within the set shall arrive at the laboratory in one of the following manners:

1. Individual potatoes in either plastic or paper bags sealed by the collector.

2. A 10-pound bag of potatoes.

b. Those samples, or portions thereof, received in a damaged condition shall be discarded and not analyzed. Condition and disposal shall be documented on the Sample Information Form (SIF).

1. If a sample bag should split open during shipment, causing a portion of the sample to come in contact with a portion(s) of another sample(s), the affected samples shall be discarded and not analyzed. The date and time received shall also be documented on the SIF.

2. The bags shall not be opened, but shall be visually inspected for any deteriorating condition (e.g., leaking of paper bag, rotting of sample apparent through plastic bag) which would make the sample inedible.

c. Each sample shall be assigned a unique laboratory identification number. This number shall be recorded in permanent non-smearing ink or waterproof stickers on the sample container and the accompanying paperwork.

d. Each laboratory shall maintain a log of samples received. Suggested methods are:

1. Each sample shall be logged into a bound notebook with ink. Mistakes shall be crossed out (one single line, no whiteout) and corrections dated and initialed. Minimum information for the logbook includes sample numbers, date, and time received (unless documented on the SIF) and recipient name/initials. Other information may include commodity type, reference to the analytical method, results, and date when results were reported.

2. Computer logs are also acceptable. The laboratory shall assure that verified hardcopies are generated on a routine basis and that electronic storage of data follows acceptable practices. Refer to USDA, AMS SOP DATA-5.

5.2 Sample Preparation

a. Potato samples may be stored in a cool, secured cabinet for a maximum of seven days prior to homogenization.

b. The analytical sample is defined as follows:

1. Large potatoes

a. Each sample shall be comprised of 10 individual potatoes or a 10-pound bag of potatoes. For samples collected individually (i.e., 10 potatoes are submitted as the sample), all potatoes shall comprise the sample.

b. For samples received as a 10-pound bag (minimum of 1/4 pound or 115 gram individual potato size), the largest 10 potatoes shall be selected to comprise the sample. The remainder shall be immediately disposed of according to the laboratory's internal procedures.

2. Small potatoes

a. Each sample shall be comprised of 20-30 individual potatoes or a 10-pound bag of potatoes. For potatoes collected individually, 2 or 3 potatoes (minimum of 1/4 pound or 115 gram total target weight) will be considered as one single serving. The laboratory will identify individual servings based on equivalent size.

b. For samples received as a 10-pound bag, the largest 20-30 potatoes shall be selected to comprise the sample. Two or three potatoes (minimum of 1/4 pound or 115 gram total target weight) will be considered as one single serving. The laboratory will identify individual servings based on equivalent size. The remainder shall be disposed of according to the laboratory's internal procedures.

c. For each sample the following preparation procedures shall be followed:

1. Hold each potato under cold running tap water and gently scrub the entire surface with a clean vegetable brush to remove any loose soil and grit. Rinse each scrubbed potato under cold running tap water for approximately 15-20 seconds to assure that all surfaces of the potato

have been rinsed and allow to drain for at least 2 minutes on paper towels on a flat surface.

2. Cut each potato in half lengthwise, reserving one half for homogenization. For each serving, the remaining half (halves for small potatoes) shall be sealed individually and labeled with the sample identification number and a designation of a-j.

3. The labeled halves shall be stored in a -40°C freezer.

4. For each sample, the reserved halves shall be mechanically chopped in a Hobart food chopper or Robot Coupe food processor just until a visually homogeneous mixture is attained. The food chopper or processor shall be thoroughly cleaned between each sample's processing according to internal laboratory SOPs.

5.3 Sample Storage

In addition to the storing of single serving subsamples, an adequate portion of the homogenized sample shall be held in reserve if re-analysis and/or confirmation is needed. This portion shall be distributed among several small containers (polypropylene or styrofoam recommended) rather than one large container. A portion shall be pre-weighed according to the analytical methodology. The laboratory internal SOP shall specify "adequate portion" and distribution.

5.4 Sample Analysis and Reporting

a. Weighing of Analytical Portion

An appropriate amount of homogenized sample shall be weighed for analysis. The laboratory internal SOP shall define the sample weight and the necessary precision which:

1. For a 50 gram sample shall not be more than +/- 0.25 grams.

2. For a 100 gram sample shall not be more than +/- 0.5 grams.

b. Sample Set Requirements

A sample set is the group of samples which are spiked individually with the designated process control and extracted on a single day along with the required QC samples. Each set shall consist of no more than 20 samples. Required QC samples per set consist of a reagent blank, matrix blank, and matrix spike(s).

The matrix spike(s) shall contain aldicarb, aldicarb sulfoxide, and aldicarb sulfone at 2xLOQ. Each sample shall be spiked with the surrogate standard, propoxur, at 5xLOQ. All components of sample sets shall be subject to the sample analytical process as detailed in the method SOPs.

c. Acceptability Criteria

1. The LOQ for each analyte (aldicarb, aldicarb sulfoxide, and aldicarb sulfone) shall be 0.02 ppm to 0.05 ppm.

2. Process control and matrix spikes shall be subject to the criteria and reaction procedures set forth in USDA, AMS, PDP SOP QC-4 "Acceptability Criteria for Process Control and Fortification Recoveries".

3. For each set the requirement for running single serving analyses shall be as follows:

a. If every sample in a set contains no detectable residues (i.e., $\geq$LOD) for any component of the aldicarb tolerance expression (i.e., aldicarb, aldicarb sulfoxide, or aldicarb sulfone), then no single serving analysis shall be required. Results shall be faxed immediately to USDA, AMS, followed by RDE transmission.

b. For each set, the sample having the highest total aldicarb (i.e., aldicarb + aldicarb sulfoxide + aldicarb sulfone) result shall undergo single serving analysis:

1. Subsamples a-j for that sample shall be removed from the -40°C freezer and homogenized individually

according to the procedures outlined above.

2. Reserve portions of subsample homogenates shall be stored in a -40°C freezer.

3. The subsamples shall be analyzed as a set with appropriate QC samples.

4. Results shall be faxed immediately USDA, AMS, followed by RDE transmission.

5.5 Sample Disposal

Samples and subsamples for each set shall be disposed of when all requirements for acceptability criteria, including applicable single serve analyses, have been met and reported via RDE to USDA, AMS. Disposal shall be documented (e.g., in the freezer log or sample log) and shall contain a minimum of date of disposal, sample number, and initials of the individual who discarded the sample.

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